IMPROVED METHODS FOR STUDYING THE CHEMICAL DURABILITY OF GLASS BY THE POWDER METHOD

BY

SUDHIR SEN

M.Sc., Calcutta University, 1940
M.S., University of Illinois, 1949

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SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
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IN THE GRADUATE COLLEGE OF THE
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R. UNIV. BIBLIOTHEEK LEIDEN

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Introduction

The American Ceramic Society tentative method No. 1 for the chemical durability test on glass requires an amount of glass of the order of 200 grams and a specific method for crushing and sieving to obtain, as far as practicable, the same surface exposed to attack. The major purpose of this work was to develop a method for assessing the chemical durability of glass which would permit the use of a small amount of sample, avoid the laborious process of crushing, sieving and recrushing as well as eliminate the uncertain factor of surface area.

It was also considered preferable, from the standpoint of interpretation, to establish methods for determining the different ions removed in a particular case rather than to measure the extent of attack by a simple titration of combined alkali[1]. Such direct methods of assessing the attack could then be used to repeat the work of Peddle[2], on the relationship of the alkalies for the maximum durability in mixed alkali-lime-silica glasses.

A few fundamental studies such as the influence of the shape of the container, influence of the ratio of the amount of sample and the amount of the attacking medium, and the effect of time and temperature were made part of this study in order to determine the extent to which these factors can influence the chemical durability measurements.

Experimental Procedure

Seven mixed alkali glasses of the composition Na_2O 0-18%, K_2O 18-0%, CaO 10%, and SiO_2 72% were used in most of this work. The durabilities of these glasses were determined following the A. C. S. tentative method No. 1 as far as the method of sample preparation and exposure. The extent of

attack in each case was assessed by the direct measurement of the ions in the extract.

The water permeability method[3] proved to be a satisfactory method for the direct measurement of surface per unit volume of randomly crushed samples of glass.

From a combined method of measuring surface with water at high temperature by the permeability method and measuring the attack by the determination of ions in the liquid collected during the same run, it was found possible to determine the chemical durability with 10 grams of glass and within a period of ½ hour.

The methods used for the determinations of ions in the extract were the following:

(1) Flame photometer method for Na_2O and K_2O.

(2) The turbidity method of Saifer and Clark[4] for CaO.

(3) The improved silico-molybdenum blue method[5] for SiO_2.

Two soda-lime-silica glasses were used in the study of the influence of the shape of the container, influence of the ratio of the amount of a sample and the amount of the attacking medium and the effect of time.

The effect of temperature was studied by the combined method of determining the chemical durability.

Discussion of Results

The combined method of chemical durability measurement on the mixed alkali glasses gave a curve with a minimum of chemical attack at the same point as that obtained by the A.C.S. tentative method No. 1.

In a series of mixed alkali glasses of composition — alkali 18%, CaO 10% and SiO_2 72% — the number of mols of total alkali extracted decreases with decrease in Na_2O content of the glass until at a ratio of about K_2O/Na_2O (by weight) 4.14/1, it reaches a minimum. This minimum is not well

marked; with further increase of K_2O only a slight increase of mols of total alkali extracted is observed. When the mols of Na_2O are in excess, the sodium exerts the controlling influence on the attack. The influence of potassium becomes evident only when K_2O is in large excess. Decrease in the extraction of CaO and SiO_2 with decrease in Na_2O content of the glass also indicate that the sodium plays the main role in the extraction.

Measurement of surface by the permeability method on different portions of the same sample of glass, collected between the same limiting screens, showed a variation of 3%. The variation of surface on samples obtained by different crushing procedures, but using the same limiting screens, was as large as 34%, thus demonstrating the great desirability of surface measurements on individual samples.

It was apparent that the calculated results of amount of attack, based on the linear relationship of the attack and the surface, would be more accurate as the time of exposure is decreased.

The log-log function of the amount of attack, N, and the time, θ, suggested by some workers, was found true for only small values of K and θ.

When water was used as an attacking medium, the relation between the amount of attack, K, and the absolute temperature, T, was found empirically to be $K = \log b/T + C$.

It was found that keeping the ratio of glass:water the same, an increase in weight of sample increases the attack upto a limit of 4-5 grams of sample; further increase gave a dilution effect. The increase in water:glass ratio also showed an increase in extraction upto a limit of 20:1; any further increase decreased the attack. The two types of vessel used, Erlenmeyer flask and round-bottomed flask, gave almost similar results.

The method of measuring surface with water at high temperature by the permeability method combined with the

direct determination of ions in the extract is a highly effective means of studying the resistance of glass to chemical attack.

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VITA

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